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**Onsite Analysis of Organic Compounds
Using Field Screening Techniques**

prepared by

**Bruce L. Ball
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presented at

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Onsite Analysis of Organic Compounds
Using Field Screening Techniques

Bruce L. Ball¹, AM

Abstract

This paper describes the use of portable wet chemistry soil field screening techniques to detect organic compounds commonly found at sites undergoing site investigations and remedial actions. Colormetric, immunoassay, and thin layer chromatography field screening methods are described, and application limitations are discussed. The methods are compared based on a variety of factors including sensitivity, specificity, accuracy, and cost. Method selection, quality assurance/quality control procedures, and regulatory acceptance of field screening methods are also discussed. Two case studies are presented illustrating the use of field screening techniques to define the spatial extent of contamination.

Introduction

Field screening methods are analytical techniques used under field conditions to characterize organic and inorganic compounds in various matrices. The numbers and types of field screening technologies have increased significantly over the past 10 years in response to the environmental industry's demand for inexpensive, simple, accurate, and rapid chemical characterization methods that can be used onsite. A large array of field screening technologies has been or is being developed.

Field screening is defined by the U.S. Environmental Protection Agency (1993) as methods that (1) provide an indication of the presence or absence of a target analyte

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or (2) provide an indication of the analytes concentration relative to a predetermined concentration. Field screening generally does not provide chemical-specific information, such as type of compound within a chemical class. Field screening techniques are tools for performing cost-effective contaminant delineation during site assessments, emergency remedial actions, remedial investigations, and remedial activities. Field screening does not replace laboratory analysis, but does decrease the number of samples sent to the laboratory for analysis.

This paper focuses on available portable wet chemistry soil field screening techniques for polychlorinated biphenyls (PCBs), petroleum hydrocarbons (BTEX, diesel, jet fuel, kerosene, heating oil), pentachlorophenol (PCPs), and polynuclear aromatic hydrocarbons (PAHs). The methods discussed do not require specialized experience to use and have a cost advantage over laboratory analysis.

Advantages/Disadvantages of Field Screening

The primary advantages of field screening compared with laboratory analysis are quicker turnaround time and lower cost. Quick turnaround time allows for rapid delineation of contamination, onsite selection of optimal boring and monitoring well locations, and segregation of clean and contaminated soil during excavation for removal and treatment. Typical laboratory turnaround times range from 1 to 5 weeks depending on the number of samples submitted and number of analyses. Field screening can provide results within 10 to 60 minutes. The lower cost of field screening allows for more frequent sampling and analysis than would otherwise be economically viable. More data points result in accurate characterization of the vertical and horizontal distribution of a contaminate at a site. Field screening costs are typically 30 to 80 percent less than laboratory costs.

The primary disadvantages of field screening are precision and accuracy of the data. Field instrumentation is generally less sophisticated and accurate than laboratory equipment, resulting in generally higher detection limits and lower precision and accuracy compared with laboratory gas chromatography (GC) analysis.

Description of Methods

Available portable wet chemistry field screening methods for organic compounds include colormetric, immunoassay, and thin layer chromatography techniques.

Colormetric Techniques

Colormetric techniques are methods that use color reagents to indicate the presence or absence of a chemical,

and generally use color charts to estimate the concentration of the chemical. Colorimetric kits currently available for soil organic analysis are manufactured by Hanby and Dexsil. Hanby kits are capable of detecting a full range of petroleum hydrocarbons, PCBs, and PAHs. Dexsil's Clor-N-Soil™ kit is for PCB analysis only.

The analysis procedures for the Hanby kits involve the addition and mixing of a color reagent and catalyst to the test tubes containing the sample extract. The hue and intensity of the sample are then compared with color photographs to determine the contaminant type and concentration. Hanby has developed a large library of photographs for a full range of aromatic compounds over a wide concentration range.

The Dexsil test is an indirect method for analyzing PCBs by measuring the total chlorine concentration in a sample. The Dexsil Clor-N-Soil™ PCB kit analysis procedures call for the addition of metallic sodium to strip the chlorine from the PCB molecule. The chlorine is then transferred to the aqueous phase and measured with mercuric nitrate and a color indicator solution. Both Hanby and Dexsil have adapted analyzers for use with their kits to provide quantitative results. The meters are relatively expensive and therefore are only economically viable for high throughput projects when more than 100 samples are to be analyzed.

The following is a list of factors that may affect the accuracy of colorimetric test results.

1. The Dexsil Clor-N-Soil™ tests cannot be used at sites suspected of being contaminated with chlorinated compounds.
2. The semi-quantitative results are subjective because sample results are compared with color charts or photographs.
3. PCBs cannot be distinguished from oil with the Hanby kits if high concentrations of oil are present in the soil.

Immunoassay Techniques

Immunoassay methodology has been used in the medical field for more than 20 years for rapid and accurate measurement of analytes such as hormones, microorganisms, and drugs. The first immunoassay kits for use in the environmental field were introduced in 1991 by EnSys. There are now four manufacturers of immunoassay kits applicable to the environmental field including EM Science (D-Tech™), EnSys (RISC™), Millipore (EnviroGard™), and Ohmicron (RAPID Assay™). Immunoassay kits are available for petroleum hydrocarbons, PCBs, PCPs, PAHs, pesticides, and ordnance analysis for water, soil, and equipment surfaces.

Immunoassay techniques employ antibodies that target specific analytes. A wide variety of immunoassay formats have been developed to allow visual or instrumental measurement of the binding reaction between the antibody and the target analyte. The antibodies are coated to either single-use polystyrene test tubes, microtiter plates, particulate systems, or magnetic particles. The analytes compete with known amounts of enzyme conjugate for a limited number of antibody binding sites. The more analytes there are in the sample, the more enzyme conjugate they will displace from the binding sites. Substrate and chromogen are added for color development. The amount of enzyme conjugate bound to the antibody is then measured with a portable spectrophotometer or reflectometer. The amount of bound conjugate is inversely proportional to the amount of analyte in the sample.

EnSys and Millipore use the same spectrophotometer in their basic kit. Ohmicron provides a more advanced and expensive programmable analyzer that automatically convert immunoassay optical readings to sample concentrations, and store sample data and calibration curves. The analyzer also include printers for automatic printout of results. EM Science has developed a low cost hand-held reflectometer that can store a maximum of 127 sample readings.

The following is a list of factors that may affect the accuracy of immunoassay test kit results.

1. Detection limits will vary for compounds within a chemical class (i.e., Aroclors). The methods are not compound specific; therefore, prior knowledge of the compound of interest is important to improve sensitivity and accuracy of the tests.
2. The immunoassay field screening methods are biased positive.
3. Ambient temperature can affect reaction rates, requiring adjustments to the incubation times.
4. The PCR sensitivity decreases for samples with petroleum concentrations in excess of 1 percent.
5. The petroleum kits can only be used for light hydrocarbons such as BTEX, diesel, jet fuel, etc.

Thin Layer Chromatography

Unlike the previous wet chemistry techniques discussed, thin layer chromatography (TLC) is not commercially available in kits. However, Friedman and Bruya (1991) have developed a list of materials required to perform analysis on 50 samples including QA/QC requirements. TLC is more difficult to perform than the colorimetric and immunoassay kits profiled. TLC is useful for field screening compounds not currently available in user friendly kits, such as heavy fraction hydrocarbons. TLC can also provide specific information such as type of

petroleum hydrocarbon, and whether the compound is a new or old spill.

TLC is a well established analytical technique that has been used for more than 40 years for organic analysis and has been adopted for use in the field by Friedman and Bruya (1991). TLC is an inexpensive technique that can be used for the analysis of samples for semi-volatile and non-volatile contaminants.

TLC is a solid-liquid chromatographic system. The liquid phase is used to carry, or physically move, the analytes from one point to another through the solid or immobile phase. The solid phase is silica gel that is coated onto a glass plate. As the analytes move, they partition between the solid and liquid phase. Analytes strongly attracted to the solid phase will remain on the solid phase longer and move slower than analytes that are not attracted.

The analyses results are visualized after the chromatography process has been stopped by placing the TLC plate in a container with iodine crystals or by using an ultraviolet (UV) light. Some compounds, such as transformer mineral oil, are not visible under UV light and can only be seen in the iodine chamber. To determine the concentration of the analytes present, the intensity of the unknown sample is visually compared with standards of known concentrations.

The following factors may affect the use of TLC techniques and their results.

1. The silica gel plates are sensitive to dirt and must be used in a relatively clean environment.
2. Results are subjective because concentrations are evaluated based on intensities relative to the intensity of standards with known concentrations.
3. Field personnel must have a basic understanding of chemistry and some laboratory experience.
4. The moderate sensitivity of TLC may limit its application in cases where low detection limits are required.

Performance Characteristics

Sensitivity

Minimum detection limits, based on manufacturer data, are listed in Tables 1 and 2. None of the tests can distinguish a specific compound within a chemical class (i.e., Aroclor 1248 cannot be distinguished from other PCBs). Different physical and chemical properties between compounds of the same chemical class will affect the sensitivity of the tests. The best sensitivity can be achieved if the target analyte is known (i.e., Aroclor 1248, diesel) and the test is modified to use target analyte standards. The sensitivity of the TLC tests is

generally higher than that of the colormetric and immunoassay tests. The skill of the analyst in spotting the TLC plates and comparing the visual presentation of the sample to standards is a critical element in establishing reliable sensitivity levels.

Weathering of the target analyte can also affect the sensitivity of the tests. Studies performed by Rittenburg (1993) and Millipore Corp. (1994) have shown that weathered samples analyzed with the immunoassay petroleum tests may give higher apparent readings compared with GC readings, due to the higher proportion of semivolatiles present following the loss of the volatile components. This factor will cause the test to have a positive bias.

Accuracy

Accuracy is defined for quantitative analysis as the ability of the test to predict the value of the analyte as determined by a GC. Accuracy is expressed as the slope and the correlation coefficient of the linear regression for split samples analyzed by field screening methods and by a GC. Accuracy for semi-quantitative results are calculated by comparing the interpreted results with the GC results. Accuracy values, based on manufacturer data, are listed in Tables 1 and 2.

Neidigh (1993) compared immunoassay PCB kits from EnSys and Millipore and reported a 97.26 percent correlation between the EnSys kits and the GC and a 98.43 percent correlation between the Millipore kits and the GC. The accuracy of the colormetric and TLC field tests can be affected by the interpretative skill of the analyst to visually compare the results of the unknown sample with standards. The closer the concentration of the unknown sample is to a standard, the better the accuracy.

Specificity

Field screening methods will not differentiate between the target analytes and other closely related compounds, but will detect their presence to differing degrees. For example, the PCP test kits will detect other phenols in addition to PCP. The specificity of some test will vary for compounds within a chemical class. For example, the immunoassay PCB kits are calibrated with a single Aroclor (1254 or 1248) and lesser Aroclors, such as 1232 and 1016, have less response to the test.

A mixture of compounds will tend to degrade the specificity of field screening tests. The TLC and the Hanby test cannot distinguish PCBs from oil. Gaskill reported immunoassay tests may be susceptible to non-specific interferences, especially nonaqueous solutions. In a comparison of the EnSys PCB immunoassay test kit with the Dexsil L2000 kit and GC, Gaskill showed that the immunoassay test consistently underestimated the PCB

concentration, when the concentration of oil in soil was greater than 2 percent and 20 ppm Aroclor 1242. At higher Aroclor concentrations, the oil had less impact on test results. The Dexsil quantitative kit results were 100 percent comparable with the GC results for all oil concentrations tested in the study.

None of the petroleum immunoassay field tests can separate hydrocarbon mixtures (BTEX versus diesel). The immunoassay test are only valid for light fraction hydrocarbons such as BTEX, diesel, and kerosene. Prior knowledge of the target petroleum compound is required for accurate concentration determination. The colorimetric and TLC tests can distinguish among petroleum products provided the products are relatively pure. Friedman and Drury have developed a catalog of TLC patterns for delineating various analytes. Hanby has also developed a library of photographs that correspond to various aromatic compounds at varying concentrations.

Biased Positives/Negatives

Biased negatives occur when the predicted concentration result is lower than the GC result. Biased positives occur when the predicted concentration result is higher than the GC result.

The Dexsil tests are susceptible to biased positives in the presences of chlorinated compounds other than PCBs. For immunoassay tests, biased positive rates typically range from 1 to 10 percent, and biased negatives are generally less than 5 percent. The Ohmicron immunoassay tests are the least susceptible to biased positives and negatives. The antibodies used for the Ohmicron method are coated to magnetic particles that have been shown (Aga and Thurman 1993) to be superior to other solid surfaces. The Ohmicron tests typically have biased positive rates of less than 3 percent and biased negative rates of less than 1 percent.

In a comparison of Millipore's and EnSys test kits, Neidigh et al. (1993) reported a 0.6 percent biased positive rate and a 2.1 percent biased negative rate out of 329 samples analyzed using the EnSys kits. Of the 64 samples analyzed with the Millipore kits, Neidigh observed 1 biased negative and no biased positives. The nature of the TLC tests limit biased negatives and positives. A biased contaminated with material or finger prints. Biased negatives can occur if the iodine development chamber is too cold.

Kit Selection

Numerous factors impact the selection of an appropriate field screening technique. Some of the factors to be considered include cost, throughput requirements,

data quality objectives, site characteristics, turnaround time, and simplicity. A comparison of factors are presented in Tables 1 and 2.

The Dexsil, EM Science, EnSys, and Hanby kits are designed for low throughput (less than 20 samples per day) and single sample analysis, but can also be used for high throughput. Millipore, Ohmicron, and TLC are designed for high throughput (greater than 20 samples per day) and for batch type analysis. Equipment and analyzer cost associated with the high throughput methods make their use as low throughput kits too expensive. EnSys and Millipore kits provide semi-quantitative results. The Ohmicron kit is designed to provide semi-quantitative or quantitative results. The Hanby and Dexsil kits can also be used quantitatively provided the optional analyzer is rented or purchased with the kit.

All of the kits, with the exception of TLC and Dexsil's semi-quantitative PCB kit, are capable of very low detection limits. The Hanby, EnSys, and TLC kits provide the most flexibility for analyzing high concentrations of the target analyte without dilutions. EnSys customizes their kits to the project requirements. The detection range of the Ohmicron method is narrow, which increases the frequency of dilutions if the kits are used quantitatively.

Other practical factors that might affect the selection of kits are batch run times and skill level required to use the kits. The colormetric kits are designed to provide single sample results in as little as 5 minutes per sample. The other field screening kits are designed to run batches of 4 to 20 samples, which increases the sample preparation and analysis time. The colormetric tests are by far the easiest test to run and require little skill or knowledge of chemistry. The TLC test are difficult to run compared with other methods and require at least some prior laboratory experience.

The chemicals used in the Hanby kit are toxic and volatile. When performing tests, special care is required to keep the chemicals out of the breathing zone and off the skin. The chemicals are also an explosive hazard if mixed with water.

Field QA/QC Procedures

Although sample collection and analysis strategies will vary, there are basic QA/QC guidelines that should be incorporated with all projects when using wet chemistry field screening analytical techniques. Manufacturers' QA/QC procedures are usually limited to requirements that are essential for the successful use of their kits. These procedures are often inadequate to meet the requirement of having defensible field screening data. The degree to which the following QA/QC procedures are incorporated with

field screening in a project is highly dependent on the quality of data that is required and the type of analysis to be performed.

Field Working Environment

The environment in which the analytical work is being performed can have a direct effect on the field screening results. Field screening should be conducted in a dry, well lighted space, whether outside or in a temporary shelter. Changing weather conditions can have an adverse affect on both the tests and skills of the analyst. The operating temperature of field screening kits vary, but are generally designed for temperatures between 60 and 100 F. Temperatures outside this range may affect the reaction rates, requiring modification of procedures. Exposure of the immunoassay test, TLC test, and electronic equipment to temperatures greater than 100 F will severely degrade the quality of the results.

Blanks

To assess the effect, if any, of the extraction solvent on the quantitation process and to check for field laboratory contamination, a blank of the extraction solvent is required with each sample batch. Blanks should always be negative for the target analyte; if they are not, the whole batch run is invalid.

Sample Duplicates

Sample duplicates, also known as splits, are analyzed to evaluate the homogeneity of a sample. Sample duplicates should be analyzed for a minimum of 10 percent of all samples field screened, regardless of data quality requirements. Any sample sent to an offsite laboratory for confirmatory analysis should be analyzed in duplicate. A wide variation in duplicate results will likely result in poor agreement with the offsite results. The precision between duplicates should be within in factor of three.

Sample Replicates

Sample replicates are multiple analysis of the sample extract. Sample replicates should be analyzed for a minimum of 10 percent of the samples, regardless of data quality requirements. Replicates provide an indication of the precision of the analytical method. Except for the colormetric tests, numerous steps are required for the immunoassay and TLC tests, thus the possibility of error is introduced. Replicates are especially important if quantitative analysis is to be performed. The precision between replicates should be less than 20 percent.

Calibration Standards and Standard Duplicates

Standards are utilized, when color charts are not used, to assist in quantifying the concentration levels in a sample. Calibration standards should be run with each batch to equalize the effect of temperature and matrix variability on the analytical process. Standard duplicates should also be run for at least one standard to check precision. The precision of the standards should be less than 20 percent.

Background Matrix Analysis

A clean sample from the site should be analyzed to document the effect of the matrix on the field screening method. If clean onsite soil is not available, other soil that is representative of the contaminated soil may be used.

Confirmation Samples

Confirmation samples should be sent to the laboratory for analysis for a predetermined percentage of field screened samples. This predetermined percentage is a function of data quality requirements, type of project, regulatory requirements, potential for interfering compounds, and experience level with the method. Confirmation samples provide an indication of the accuracy of the field screening results. As a minimum, 5 to 10 percent of the samples should be sent to the laboratory if semi-quantitative analysis is to be performed and data quality requirements are low. For quantitative analysis and high data quality objectives, 10 to 20 percent of the samples should be sent for analysis. Confirmation samples should include a full range of samples from the detection range, including non-detects, samples near the threshold requirements, and "hot" samples.

Regulatory Acceptance

Some field screening methods have been approved as draft methods for inclusion into the third update of *Test Methods for Evaluating Solid Waste, SW-846*. EnSys and Millipore's PCB immunoassay kits have been excepted for inclusion in draft Method 4020. EnSys' PCP method was included as draft Method 4010 in the second update of *Test Methods for Evaluating Solid Waste, SW-846*. EnSys' petroleum and PAH methods have been accepted for inclusion into draft Methods 4030 and 4035, respectively. Millipore, EM Science, and Ohmicron are currently seeking inclusion of their methods into the draft Methods. Also, Hanby and Dexsil are seeking EPA approval of their methods for incorporation into SW-846. TLC is not currently under consideration for inclusion as a field screening method under SW-846.

Case Studies

The following two case studies profile the applications of field screening methods at a series of site investigations of electrical substations and at a remedial action involving the removal of contaminated sediments in a creek.

Electrical Substation Site Assessments

Environmental assessments were performed for two active and seven inactive electrical substations located within the Tacoma, Washington metropolitan area on residential, commercial, industrial, military, and Superfund properties (BVWST 1993). The two active substations were investigated to evaluate previous cleanup actions. The seven inactive substations were to be sold for redevelopment. During the operational life of the substations, soil, concrete, and equipment surfaces had become contaminated with PCBs and mineral oil.

PCB cleanup levels for the State of Washington are 1 ppm for residential property, 14 ppm for commercial property, and 18 ppm for industrial property. The mineral oil cleanup concentration is 200 ppm.

Millipore EnviroGard™ PCB kits were selected for use semi-quantitatively at a 1-ppm threshold. TLC was used semi-quantitatively for mineral oil analysis. More than 900 samples were collected and analyzed for PCBs and mineral oil at a rate of 40 to 80 samples a day. Confirmation samples were collected and sent to a laboratory for approximately 10 percent of field screened samples. The correlation rate yielded a 100 percent accuracy rate for PCB concentrations less than 1 ppm and a 93 percent accuracy rate for PCB concentrations greater than 1 ppm. The results of the immunoassay kit yielded a biased positive rate of 4.6 percent and no biased negatives. The correlation rate for the TLC mineral oil analysis was 78 percent, with the concentration of 11 percent of the samples overestimated and 11 percent underestimated.

Figure 1 illustrates how the field screening results were used to determine the spacial extent of PCB and mineral oil contamination at one of the substations. Similar site maps were developed for all of the substations with contamination and used to direct the remediation effort. Use of field screening saved the client \$20,000 in laboratory fees or 15 percent of the project's cost.

Creek Remedial Action

Joint caulking containing up to 300,000 mg/kg of PCBs was used in a buried 65.6 million gallon reinforced concrete reservoir when the reservoir was constructed 30 years ago in Northern California (BVWST 1994). During

cleaning of the reservoir, some of the deteriorated caulking material was washed into the overflow/drain line. The reservoir overflow/drain line terminates at an energy dissipator, where discharged water is released into a pristine creek. The creek flows through a historical park and garden and discharges into a storm sewer at the southern boundary of the property before discharging into the San Leandro Creek.

Initial sampling of the creek indicated the presence of PCBs of 0.08 to 46 mg/kg at the dissipator and as high as 320 mg/kg in the creek bed. The initial remedial action included removal of sediment to a depth of 1 foot, 50 feet upstream and 500 feet downstream of the energy dissipator. Confirmation sample results were received 3 weeks later indicating PCBs were still present above the action level of 0.1 mg/kg. Subsequent sampling indicated the linear extent of PCBs in the creek bed extended 1,800 feet downstream of the dissipator. PCB field screening was instituted into the second phase of the remedial action to provide onsite results, facilitate soil segregation and removal, and to minimize the number of samples sent to the laboratory. To maximize analysis and removal efficiency, the creek bed was divided into 50-foot sections and 10-foot subsections. A seven-point hexagonal sampling grid was used to collect composite samples for field screening from 10-foot lengths of the creek. If the results were above threshold levels, a 1-foot lift of soil was removed and the section resampled. This process continued until field screening indicated the section was cleaned. If the field screening results were below threshold requirements, the sample was sent to the laboratory for analysis. Over 2,300 samples were field screened. A total of 152 confirmation samples were collected from the creek bed and 290 confirmation samples from the creek banks. The biased negative rates were 0.6 percent for Aroclor 1248 and 2.5 percent for Aroclor 1254. Use of the field screening saved the client in excess of \$200,000 in laboratory, mobilization, and downtime costs, and allowed the client to quickly remediate the site.

Conclusions

Use of field screening methods in conjunction with confirmatory laboratory analysis can result in accurate delineation of contaminant plumes at a significant cost savings in site characterization efforts and remedial actions. Analytical results can be obtained within 10 to 60 minutes, allowing for field decisions regarding boring placement or soil segregation. Field screening methods need to be selected based on site characteristics, data quality objectives, and regulatory requirements.

Table 2. Immunoassay Techniques

Parameters (1)	Immunoassay			
	EM Science D-Tech™ (2)	EnSys RISC™ (3)	Millipore Enviro Gard™ (4)	Ohmicron RaPID Assay™ (5)
Sensitivity (ng/kg) ^{1,2}				
Petroleum	1.0	10	2.0	NA
PCB	0.5	0.4	0.5	0.5
PCP	UD	0.5	0.05	0.1
PAH	UD	1	1.0	NA
Accuracy % ²	96-99	93-95	92	91
Skill Level ³	Low	Medium	Medium	Medium
Samples per Kit ⁴	4	4	12	20 or 80
Results ⁵	S	S	S	S, Q
Throughput Options	Single/Batch	Single/Batch	Batch	Batch
Batch Run Time ⁶ (min)	20/30	30/45	50	60
Analysis/Extraction Kit Cost ⁷ (\$)	127	150-225	388	600
Cost per Sample (\$)	31	38-56	33	30
Equipment ⁸ Cost (\$)	NR	315-780	1,570	1,503
Analyzer ^{8,9} Cost (\$)	299	935	839	800-3,985

¹ Sensitivity will vary depending on target analyte within listed chemical class.

² Information provided by manufacturer.

³ Training required: Low = <2 hour, Medium = 2 to 6 hours, High = >6 hours.

⁴ Actual number of samples per kit may vary with QA/QC requirements.

⁵ S = semi-quantitative, Q = quantitative.

⁶ Analysis time based on single and/or multiple sample analysis.

⁷ Actual cost may vary.

⁸ Purchase cost; rental and loaner programs are offered by some manufacturers.

⁹ Analyzer costs will vary depending on degree of sophistication required.

NR None required. NA Not available. UD Under development; available in 1994 or 1995.

Table 1. Colormetric and TLC Techniques

Parameter (1)	Colormetric		Thin Layer Chromography (4)
	Harby (2)	Dexsil Clor-N-Soil™ (3)	
Sensitivity (mg/kg) ^{1,2}			
Petroleum	1.0	UD	50
PCB	5.0	5.0	5.0
PCP	NA	NA	5.0
PAH	0.05	NA	5.0
Accuracy % ²	99	99	>80
Skill Level ³	Low	Low	High
Samples per Kit ⁴	30	20	50
Results ⁵	S	S,Q	S
Throughput Options	Single/Batch	Single/Batch	Batch
Batch Run Time ⁶ (min)	10/45	10/20	75-180
Analysis/Extraction Kit Cost ⁷ (\$)	1,195	10	935
Cost per Sample (\$)	40	10	19
Equipment ⁸ Cost (\$)	NR	NR	200
Analyzer ^{1,9} Cost (\$)	3,995 (opt)	3,500 (opt)	250

¹ Sensitivity will vary depending on target analyte within listed chemical class.

² Information provided by manufacturer.

³ Training required: Low = <2 hour, Medium = 2 to 6 hours, High = >6 hours.

⁴ Actual number of samples per kit may vary with QA/QC requirements.

⁵ S = semi-quantitative, Q = quantitative.

⁶ Analysis time based on single and/or multiple sample analysis.

⁷ Actual cost may vary.

⁸ Purchase cost; rental and loaner programs are offered by some manufacturers.

⁹ Analyzer costs will vary depending on degree of sophistication required.

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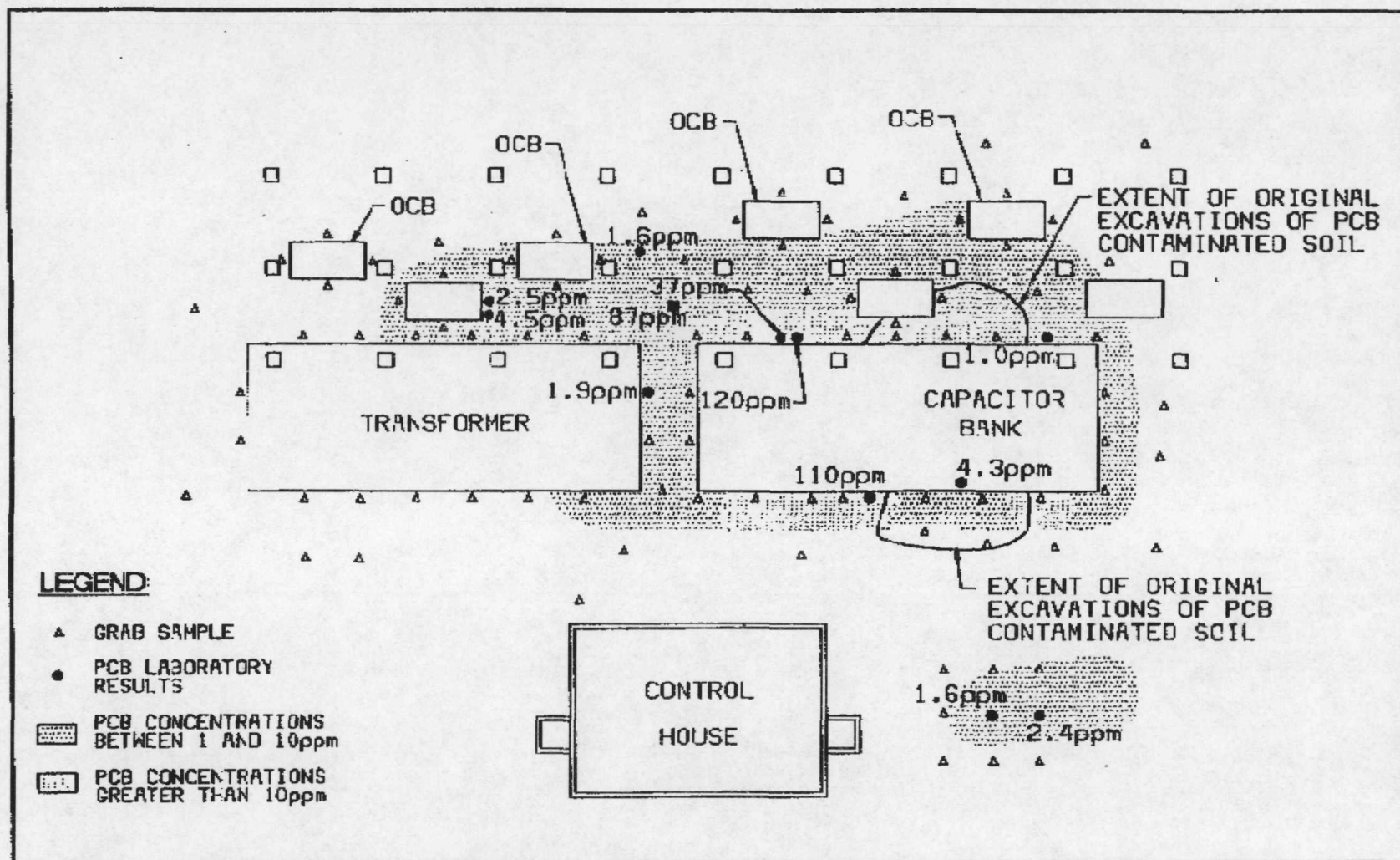


Fig. 1. Field Screening Results at an Electrical Substation

Appendix I. References

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

JAN 11 1996

SR-6J

CERTIFIED MAIL
RETURN RECEIPT REQUESTED
AND FACSIMILE

Mr. Ronald Frehner
Project Coordinator - ACS NPL Site
Conestoga-Rovers & Associates
1801 Old Highway 8, Suite 114
St. Paul, Minnesota 55112

RE: Review of Responses to U.S. EPA Comments, and Modifications to Third Draft Dewatering/Barrier Wall Alignment Pre-Design Work Plan (December 15, 1995); Approval with Modifications of new SOPs (December 15, 1995); and Approval with Modifications of Requested Modification to the Work Plan regarding Immunoassay Methodology and SOP (January 9, 1996); American Chemical Services NPL Site, Inc. Griffith, Indiana

Dear Mr. Frehner:

The United States Environmental Protection Agency (U.S. EPA) and the Indiana Department of Environmental Management (IDEM) have reviewed the responses to comments and revisions for Dewatering/Barrier Wall Alignment Pre-Design Work Plan and SOPs dated December 15, 1995. U.S. EPA hereby approves the SOPs with the enclosed modifications. Respondents must address these modifications prior to the startup of field work. Replacement pages must also be submitted prior to the startup of field work.

As you know, on November 22, 1995, the U.S. EPA approved with modifications the revised Dewatering/Barrier Wall Alignment Pre-Design Work Plan contingent on approval of the Standard Operating Procedures (SOPs). The Work Plan was submitted by Montgomery Watson for the American Chemical Services, Inc., National Priorities List (NPL) Superfund Site located in Griffith, Indiana (ACS Site), in accordance to the Unilateral Administrative Order



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(UAO) (Docket No. V-W-95-C-260) issued by U.S. EPA on September 30, 1994. The responses to comments, revised Work Plan and new SOPs were received by U.S. EPA on December 15, 1995.

In addition, based upon a recent conversation with Montgomery Watson, U.S. EPA learned that Respondents planned to use the Ohmicron PCB immunoassay method in lieu of the Ensys PCB immunoassay method. U.S. EPA suggested that Respondents submit a request for a modification prior to implementation since the Ensys method had already been approved for use by U.S. EPA. [Note that paragraph V.35 of the UAO states . . . [a]ny noncompliance with the approved . . . Workplan shall be a violation of the Order.]

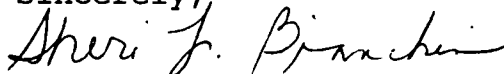
U.S. EPA received by facsimile a request for a revision to the Work Plan on January 9, 1996, to use the Ohmicron PCB immunoassay method in lieu of the Ensys PCB immunoassay method. U.S. EPA is not opposed to the use of the Ohmicron method since, just as the Ensys method, it is based upon proposed EPA SW-846 method 4020. However, the Ohmicron kit has not yet been validated by U.S. EPA to show equivalency with the EPA immunoassay method; hence, it may only be used for this project if field validation is employed throughout the project. Therefore, U.S. EPA hereby approves the requested modification to use the Ohmicron method with the enclosed modifications.

U.S. EPA would like to point out however, it is unacceptable that Respondents have made a pattern of making unrequested modifications (i.e., unilateral modifications.) The UAO not provide for unlimited changes to the deliverables. It is wasteful of all our resources to entertain modifications unless unanticipated problems are encountered. Specifically, paragraph V.32. of the UAO states . . . [s]ubmission of an amended workplan or other deliverable which fails to incorporate all of EPA's required modifications, or which includes other unrequested modifications, shall . . . constitute noncompliance with this Order.

U.S. EPA would like to stress that U.S. EPA and IDEM have expended many resources on this project to get an approvable Work Plan and would hope that Respondents would be more mindful of the process set out in the UAO in the future.

If you have any questions, or require clarification, you may reach me at (312) 886-4745.

Sincerely,



Sheri L. Bianchin,
Remedial Project Manager
Office of Superfund
Remedial Response Section #3

Enclosure

Enclosure

Review of Responses to U.S. EPA Comments and Modifications to Third Draft Dewatering/Barrier Wall Alignment Pre-Design Work Plan (December 15, 1995); Approval with Modifications of new SOPs (December 15, 1995); and Approval with Modifications of Requested Modification to the Work Plan regarding Immunoassay Methodology and SOPs (January 9, 1996);
American Chemical Services, Inc., NPL Site
Griffith, Indiana

Specific Comments

1. Page 2, Paragraph 3, third sentence.

Replace "GPCS" with "PGCS".

2. Page 8, paragraph 1, sixth sentence.

It appears that the sentence should read "The locations of the test cells are shown in the Pre-Design Work Plan." However, stating that these locations are shown in the Pre-Design Work Plan is not adequate. Include a map showing the approximate locations of the pilot test cells in the Dewatering/Barrier Wall Alignment Work Plan.

3. Figures 2 and 4.

Figures 2 and 4 must be revised so that the dashed line that identifies the "approximate extent of contaminated soil" includes SB53 and AP-65 within its limits. As stated in the U.S. EPA's Specific Comment 12, both soil probe SB53 and auger probe AP-65 contained oily waste.

4. Appendix D, Field Gas Chromatography SOP.

- A. Section D, item 3 and Section E, item 3.

It is recommended that Surrogates, *a,a,a*-Trifluorotoluene and 1,4-Dichlorobutane, be added to the samples to monitor system and method performance.

- B. Quality Control.

It is recommended that the Calibration Check Standard be analyzed at the beginning and the end of the day, in addition to after every 10 or fewer samples.

- C. Table 1.

The reporting limits presented in the table are for water not soil. The Reporting Limits for Low Soils and Medium/High Soils should be given, since this method is used for Soils. Revise the table to include approximate

reporting limits for soil. If necessary, assume a sample weight of 5 grams in the calculation.

5. Appendix G, Method 9100.

Designate which sections of this SOP are applicable in this project. Section 2.11, Leachate conductivity using laboratory methods, is probably pertinent. Discuss whether Section 3.0 **Field Methods** are applicable.

6. SOP for Immunoassay Methods.

General Comments

- A. U.S. EPA document "A User's Guide to Environmental Immunochemical Analysis" has been previously provided to you; it may still be useful for your consultation.
- B. Discuss which sections and pages of the manufacturers users guide are applicable. In addition, as well as providing the manufacturers users guide, provide a QA/QC section which is project specific. The revisions to the SOP should also be made in the next revision of the QAPP. This comment was previously provided to in the disapproval letter of July 21, 1995, and must still be addressed.
- C. High moisture content will affect sample extractability. Hence, it is recommended that the samples be dried prior to the analysis such as by using sodium sulfate.
- D. It is important to stress that it is U.S. EPA's position that the kits yield screening-level data only

Specific Comments on RaPID Assay^R Environmental User's Guide
OHMICRON

- A. Since Ohmicron's PCB kit has not yet been validated by U.S. EPA, then it may only be employed for this project if field validation is used throughout the project. The following guidance document should be consulted to establish what is required for field validation: "Immunoassay Methods for SW-846: Recommended Format and Documentation for New Submittals" (July 1995). If additional information is needed, you may contact U.S. EPA's Methods Information Communication Exchange Hotline at (703) 821-4690.

B. Section 3 - Quality Control and Sampling Plans

The Level QA2 requirements, pgs 3.4 and 3.5, should be followed, and the other **Ohmicron** recommendations given in Section G., pg 3.5, considered.

C. Section 4 - Selecting a Cutoff Concentration

Should the project decide to employ a cutoff concentration, other than the 10 ppm PCB, specify the cutoff concentration, and complete the WORKSHEET form on pg 4.5.

D. Section 4 - Selecting a cutoff Concentration

Specify the calibration method used for this analysis, most likely, METHOD 2 - CALIBRATING WITH A SITE SPECIFIC REFERENCE MATERIAL, pg 4.4. Identify the calibration standard, **Aroclor 1248**, probably.

E. Section 17 - Trouble Shooting

There is a duplicate page 17.2 preceding Section 18.